Application #: 10/663,117

SPECIFICATION

TITLE OF INVENTION

Process for forming micro-fiber cellulosic nonwoven webs from a cellulose solution by melt blown technology and the products made thereby

Ron (Rongguo) Zhao, Eckhard Schwarz
Biax Fiberfilm Corporation
N992 Quality Drive Suite B, Greenville, WI 54942

CROSS-REFERENCE TO RELATED APPLICATIONS

(Not applicable)

STATEMENT REGARDING FEDERARALLY SPONSORED RESEARCH OR DEVELOPMENT

(Not Applicable)

REFERENCE TO SEQUENCE LISTING, A TABLE, OR A COMPUTER PROGRAM LISTING COMPACT DISC APPENDIX

(Not applicable)

BACKGROUND OF THE INVENTION

1. Field of the invention

[0001] This invention relates to a process of melt blowing a cellulose solution through a concentric melt blown die with a plurality of spinning nozzles to form cellulosic microfiber webs with different web structures.

2. Description of the prior art

[0002] Cellulosic fibers are man-made fibers regenerated from a proper cellulose solution (dope) with different techniques. As an example, Lyocell fiber is one of the regenerated, man-made cellulose fibers. It is traditionally made by a dry-jet-wet-spinning process, where the cellulose solution of a solvent, such as N-methyl morpholine N-oxide, is extruded through a spinneret to form filaments. These filaments travel a short distance in air (the dry-jet), then get into a coagulation bath for regeneration. A proper mechanical pulling force is applied onto the regenerated fibers to attenuate the fiber in the "dry-jet" section. Regenerated fibers then go through a series of washing/finishing baths and drying units to form final products in the form of continuous filaments or short fibers. U.S. Pat. Nos. 4,142,913; 4,144,080; 4,211,574; 4,246,221, and 4,416,698 and others described the details of this process.

[0003] Jurkovic et al., in U.S. Pat. No 5,252,284 and Michels et al., in U.S. Pat. No. 5,417,909 deal especially with the geometry of extrusion nozzles for spinning cellulose dissolved in NMMO. Brandner et al., in U.S. Pat. No. 4,426,228, is exemplary of a considerable number of patents that disclose the use of various compounds to act as stabilizers in order to prevent cellulose and/or solvent degradation.

[0004] Zikeli et al., in U.S. Pat. Nos. 5,589,125 and 5,607,639, direct a stream of air transversely across strands of extruded lyocell dope as they leave the spinnerets. This air stream serves only to cool and does not act to stretch the filaments. French laid open application 2,735,794 describes formation of lyocell fibers by a process of melt blowing. However, these are highly fragmented microfibers useful principally for production of self bonded non-woven webs.

[0005] US Pat. 6,306,334 teaches a process using much larger sectioned spinning orifices compared with the above referenced technologies enabling a higher dope throughput per orifice to minimize tendency for orifice plugging problem. Although Example of this patent described a single orifice melt blown die with air delivered from both sides of the die through parallel slots at an angle of 30 degree, it failed to teach more details of a die with multiple orifices, such as that how the orifices are arranged, and how the air applied to extruded filaments. Due to the unique characteristic of cellulose-NMMO solution and complexity of MB technology, it is uncertain that if the same results from a single orifice MB die could be obtained from a multiple orifice MB die.

SUMMARY OF THE INVENTION

[0006] The present invention is directed to a process of melt blowing a cellulose solution through a concentric melt blown die with multiple rows of spinning nozzles to form cellulosic microfiber webs with different web structures. The term of "cellulose" as used here should be understood as either cellulose from natural resources or a synthetic polymer blend with cellulose. The term of "die" is often used as the term of "spinneret" in this invention. The term of "concentric melt blown die" refers to an apparatus described in U.S. Pat. No 5,476,616 with the hot air nozzles concentric with the polymer spinning nozzles and the air flows parallel with the polymer filaments near the exits of the nozzles.

[0007] The cellulose solution is extruded out through each spinning nozzle at a proper temperature (ranging from 80 to 140°C) and a proper throughput. The extrudates are attenuated quickly by high velocity hot air jets from a few hundred micrometers in diameter to a few micrometers in diameter within a few centimeters from the nozzle exits. These microfibers are collected on the surface of a moving collecting device, which can be either a drum collector or a flat screen collector. A set of jets of solvent/non-solvent mixture shoots from a series of fine orifices/nozzles on the flying fibers and the collected web. The term of solvent used in the present invention refers to NMMO, dilute caustic soda, phosphoric acid, mixture of liquid ammonia/ammonia thiocynate and others. The term of "non-solvent" used here refers to water, alcohol ($C_nH_{2n+1}OH$, $n \leq 10$), and/or water/alcohol/solvent solutions. The term "water" is often used as the term of "non solvent" in this invention. Depending on the position and angle of the non-solvent jet, the amount of non-solvent applied, and other factors, the resultant cellulose microfiber nonwoven web exhibits different characteristics. The jets of solvent/non-solvent solution serves two functions in this process, coagulating (fully or partially) the filaments and hydro-entangling the filaments to form webs.

[0008] The final cellulose microfibers have an average fiber diameter ranging from 1 micrometer to 20 micrometer with a relatively broad fiber diameter distribution

[0009] Spinning nozzles have an inside diameter in the range of 0.005-0.050 inch with a length/diameter (L/D) ratio in the range of 40-300. Under proper operation conditions, the resultant melt blown web is free of "shot", a defect in the form of glob of polymer which is significantly large than the fiber. Fibers produced by the method of this invention possess desirable crimps.

[0010] There is an object of the present invention to provide a method of forming cellulose microfiber nonwoven fabrics from a solution of a cellulose solvent, such as NMMO, by utilizing melt blown technology with concentric multiple-row spinning nozzles

[0011] It is an additional object to provide a method for making the said nonwoven web without additional processes, i.e. carding, web forming, and bonding.

[0012] It is a further object to provide a method for making cellulose nonwovens with different web structures.

[0013] It is another object to provide a method for forming a biodegradable nonwoven web.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] The foregoing aspects and many of the attendant advantages of this invention will become more readily appreciated as the same becomes better understood by reference to the following detailed description, when taken in conjunction with the accompanying drawings, wherein:

[0015] FIG. 1 is a schematic view of a melt blowing spinneret with multiple rows of spinning nozzles and concentric air jets used with the invention.

[0016] FIG 2 is a schematic bottom view of part of the melt blowing spinneret in FIG 1.

[0017] FIG. 3 is a diagram of the equipment used with the above melt blowing spinneret.

[0018] FIGS. 4, 5 and 6 are optical micrographs of melt blown Lyocell nonwoven at 40X, 100X and 400X, respectively.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0019] The process of the present invention is suitable to various cellulose solutions. The solvent includes NMMO, dilute caustic soda, phosphoric acid, mixture of liquid ammonia/ammonia thiocynate and others. The ways making a solution of the cellulose are known to the art, as reported by Petrovan, Collier, and Negulescu in "Rheology of Cellulosic N-Methymorpholine Oxide Monohydrate Solutions of Different Degrees of Polymerization" (Journal of Applied Polymer Science, Vol 79, 396-405 (2001)), by Albrecht in "Lyocell Fibers" (Chemical Fiber International, Vol 47, 298-304 (1997)), by Luo in U.S. Pat. No 6,306,334 B1, and by Liu, Cuculo, Smith in "Diffusion competition between solvent and nonsolvent during the coagulation process of cellulose / ammonia / ammonium thiocynate fiber spinning system" (Journal of Polymer Science Part B: Polymer Physics Vol 28, Issue 4 , Pages 449 – 465 (1990))

[0020] Reference to FIG. 1, it shows a schematic view of a melt blowing spinneret .The cellulose solution is supplied by an extruder and/or metering pump

through the cavity 2 of the distribution plate 1 to the nozzle plate 4. The plate 4 includes a gas cavity 9, which is supplied with hot compressed air or other fluids from 3. The baffle plates 8 divert the gas stream and force the gas through the gas distribution plate 5 and the air holes 13 of the top plate 7. Plates 5 and 7 are separated with a spacer 6. The hot air jets 11 reach a sonic velocity at the air hole exit, even at supersonic levels depending on the spinneret geometry and the processing conditions. The high velocity gas accelerates and attenuates the exiting cellulose NMMO solution 12 from the nozzles 10 to form fine fibers 12a. The nozzles, made of high quality stainless steel, have a length ranging from 0.5" to 3", and inside diameter (I.D.) ranging from 0.005" to 0.050", and preferably, a length from 1" to 2" and an I.D ranging from 0.009" to 0.020". The spacing of the nozzles is between 0.045" to 1.0", and preferably, between 0.050" to 0.2". The length of the nozzle sticking out of the top plate is between –0.005" to 1", and preferably, between 0.050" to 0.220". Figure 2 shows a portion of the spinneret bottom view.

[0021] The present process produces significantly more filaments per inch spinneret compared to the process disclosed in U.S. Patent No 6 306, 334 B1 and U.S. Patent No 6,358,461 B1, where a melt blowing die of single row of spinning holes employed.

[0022] FIG. 3 shows an example of detailed melt blowing process of this invention. The multiple row filaments of the cellulose solution are attenuated from a few hundred micrometers in diameter to a few micrometers in diameter within a short distance from the spinneret. With the high velocity air jets 11, these molten microfibers 12a are blown onto a paternally perforated moving surface 14, which is located 3 inch to 50 inch from the spinneret. At a point in the spinline, two sets of jet heads 15 shoot high pressure/speed hydro jets 16 onto the fiber/air stream with an angle 17 to coagulate/regenerate and entangle the cellulose microfibers. At least another set of jet heads 19 shooting hydro jets 20 is used down stream to enhance the integrity and properties of nonwoven web. The term of "hydro jet" in the present invention refers to jets of pressured liquid or mixture of liquid/air. Through a set of cartridge filters 43 and transfer lines 18, these hydro-jets 16 and 20 are supplied with circulating solvent/non-solvent solution 26 from the first regenerating bath 27 by a high-pressure pump 44. The solvent/non-solvent solution applied to the nonwoven web will return to the first

generating bath with the help of the gutter 42. The hydro-entangled web 22 guided by a roller 21 enters into the nip of pressure roller 23 and driving roller 24 travels in the first bath 27 for major regeneration. The web 22 folds in layers on slow-moving conveyer screen 28 submerged in bath 27. The conveyer screens 28 and 28a are supported by rollers 29 and 29a. The layered web 25 is submerged in solution 26 for at least 30 seconds, then enters into the nip of pressure roller 31 and driving roller 32 for liquid removing. The squeezed web folds to form web layers 33 onto the conveyer screen 28a in the bath 34 for further regeneration in fresh water. After at least 25 seconds, the web comes out of the bath 34, gets washed with fresh water sprays 35, and goes through the nip of 21a and pressure roller 36. The washed and squeezed web travels with the collector surface 14 through a zone heated with heater 37 and vacuumed with a vacuum duct 38 and vacuum head 39. The dried Lyocell fiber nonwoven web is winded up as roll 41 on the surface of the collector. At least one gutter 40 is attached to the vacuum duct to guide the penetrated solution back to the nonwoven web. The solution in bath 27 and bath 34 is stirred constantly by helical stirrers 43.

[0023] The optical micrographs shown in FIGS. 4-6 are of Lyocell fibers made by the process of the present invention. The average fiber diameter is about 5 ~15 micrometers.

EXAMPLE 1

[0024] A ¾ inch extruder is fed with a NMMO solution comprising 10.5% by weight cellulose, 77.5% by weight of NMMO and the rest is mainly water. The solid solution are in the form of pellet of 0.05" ~0.08" in size. The feeding hoper is filled with Argon gas to prevent moisture takeup. The cellulose has an average degree of polymerization from 330 ~360.

[0025] The extruder has three heating zones and the temperatures were set as 165°F (Zone 1, near the feeding hoper), 210°F, 230°F, respectively. The molten solution was forced into the body of a 5-inch-2-row spinneret, with 126 spinning nozzles (I.D. =0.009") and protruding length of 0.1915". The solution temperature and pressure at the spinneret were kept in 230°F and 600 PSI, respectively. The air temperature and pressure

in the spinneret were held at 250°F and 15PSI respectively. The solution throughput was about 0.16 gram/nozzle/min.

[0026] The attenuated microfibers are deposited on a perforated rotating drum right after contacted with the hydro needling jets. These strong needling jets serve as a pre-coagulation means and a fiber entangling means. The web goes through another set of hydro needling jets for better mechanical bonding and regenerating. The well bonded web is regenerated, washed, post-treated, and air dried.

EXAMPLE 2.

[0027] A 1 inch extruder is fed with a NMMO solution comprising 14% by weight cellulose, 76% by weight of NMMO and the rest is mainly water. The solid solution are in the form of pellet of 0.05" ~0.08" in size. The feeding hoper is filled with Argon gas to prevent moisture takeup. The cellulose has an average degree of polymerization of 670.

[0028] The extruder has three heating zones and the temperatures were set as 185°F (Zone 1, near the feeding hoper), 230°F, 250°F, respectively. The molten solution was forced into the body of a 5-inch-2-row spinneret, with 63 spinning nozzles (I.D. =0.020") and protruding length of 0.180". The solution temperature and pressure at the spinneret were kept in 250°F and 860PSI, respectively. The air temperature and pressure in the spinneret were held at 270°F and 10PSI respectively. The solution throughput was about 0.8 gram/nozzle/min.

[0029] The inventors have herein described the best present mode of practicing their invention. It will be evident to others skilled in the art that many variations that have not been exemplified should be included within the broad